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METHOD FOR MANUFACTURING ULTRA-FINE POLYESTER FIBERS

[Kyokusai Poriesuteru Seni No Seizoho]

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UNITED STATES PATENT AND TRADEMARK OFFICE

Washington, D.C.

May 2003

Translated by: Schreiber Translations, Inc.

Country : Japan

Document No. : Sho 55-93816

Document Type : Kokai

Language : Japanese

Inventor : Michiaki Hagiwara, Harayu Ogasa,
and Hitomi Tsuji

Applicant : Unitika Ltd.

IPC : D 01 F 6/62

Application Date : January 10, 1979

Publication Date : July 16, 1980

Foreign Language Title : Kyokusai Poriesuteru Seni No
Seizoho

English Title : METHOD FOR MANUFACTURING ULTRA-
FINE POLYESTER FIBERS

Specification

1. Title of the invention

Method for Ultra-Fine Polyester Fibers

2. Claims

1. A method for manufacturing ultra-fine polyester fibers, characterized by the fact that in a method for manufacturing ultra-fine fibers by setting the amount Q (g/min) of discharge for each hole of a spinneret to 0.15 g/min and a drawing speed of $18 \times 10^3 \times Q$ m/min or more in melt-spinning polyester, the following conditions A and B are met.

A: Use of a spinneret in which spinneret holes are arranged in a ring shape so that the spinneret hole diameter (D) may be 0.20 mm or less and the value of K being prescribed by the equation (1) may be 0-0.25.

$$K = D2 - D1/D1 \quad (1)$$

(D1 and D2 are minimum and maximum hole array diameters of the holes of the spinneret.)

¹ Numbers in the margin indicate pagination in the foreign text.

B: In an area within 10 cm right under the spinneret, a gas with an amount of flow of M (Nl/min) that meets the equation (2) from the outer periphery of the spinneret toward the center is blown, and the atmosphere temperature $T(^{\circ}\text{C})$ in the vicinity of the spinneret is set to the range of the equation (3).

$$\sqrt{V}(5 + \sqrt{6H - 200})/16.5 \leq M \leq \sqrt{V}(\sqrt{70H + 3500} - 30)/16.5 \quad (2)$$

(V is the drawing speed of the spinning yarns (m/min), and H is the number of holes of the spinneret and is $H \geq 14$.)

When $0 \leq L \leq 5$,

$$(50\eta - 63)L^2 + (331 - 300\eta)L + 250\eta - 70 \leq T$$

$$T \leq 130 + (5 - L)(50\eta - 34)$$

When $5 \leq L$,

$$T \leq 130 \quad (3)$$

(η is a relative viscosity of the polyester, and L is a distance from the spinneret (m).)

3. Detailed explanation of the invention

The present invention pertains to a method that economically and efficiently manufactures completely continuous ultra-fine filament yarns with high qualities without end breakage, fuzz, and fusion by an ordinary melt-spinning method from polyester.

Ultra-fine fibers are used in synthetic papers, filters, /2 artificial leathers, sweats for clothing, etc. These fibers have recently been markedly developed, and the manufacture, application, research, and development of the ultra-fine fibers have been actively carried out. As conventional methods for manufacturing the ultra-fine fibers, a peeling-off composite fiber [illegible] method, a method for removing sea components of sea-island type fibers, etc., have been proposed and industrialized. However, in these methods, there were various problems in terms of economical efficiency, operability, and yarn performances. An attempt of manufacturing the ultra-fine yarns with a single yarn size of 1 denier or less by an ordinary melt-spinning method was also made, however ultra-fine yarns, especially ultra-fine yarns with a single yarn size of 0.5 denier or less could not be manufactured with good operability due to the relationships such as surface tension of the spinning polymer.

Accordingly, these inventors researched the above problems in earnest to manufacture high-quality completely continuous ultra-fine polyester filament yarns with good operability by an ordinary melt-spinning method in terms of economical efficiency and yarn quality performances. As a result, the present invention was achieved.

In other words, the present invention is a method for manufacturing ultra-fine polyester fibers characterized by the fact that in a method for manufacturing ultra-fine fibers by setting the amount Q (g/min) of discharge for each hole of a spinneret to 0.15 g/min and a drawing speed of $18 \times 10^3 \times Q$ m/min or more in melt-spinning polyester, the following conditions A and B are met.

A: Use of a spinneret in which spinneret holes are arranged in a ring shape so that the spinneret hole diameter (D) may be 0.20 mm or less and the value of K being prescribed by the equation (1) may be 0-0.25.

$$K = D_2 - D_1/D_1 \quad (1)$$

(D_1 and D_2 are minimum and maximum hole array diameters of the holes of the spinneret.)

B: In an area within 10 cm right under the spinneret, a gas with an amount of flow of M (Nl/min) that meets the equation (2) from the outer periphery of the spinneret toward the center is blown, and the atmosphere temperature $T(^{\circ}\text{C})$ in the vicinity of the spinneret is set to the range of the equation (3).

$$\sqrt{V(5 + \sqrt{6H - 200})}/16.5 \leq M \leq \sqrt{V(\sqrt{70H + 3500} - 30)}/16.5 \quad (2)$$

(V is the drawing speed of the spinning yarns (m/min), and H is the number of holes of the spinneret and is $H \geq 14$.)

When $0 \leq L \leq 5$,

$$(50\eta - 63)L^2 + (331 - 300\eta)L + 250\eta - 70 \leq T$$

$$T \leq 130 + (5 - L)(50\eta - 34)$$

When $5 \leq L$,

$$T \leq 130 \tag{3}$$

(η is a relative viscosity of the polyester, and L is a distance from the spinneret (m).)

Also, in the present invention, the relative viscosity η of the polyester shows a value measured at a concentration of 0.5 g/100 ml and 20°C, using an equal weight mixture of phenol and ethane tetrachloride as a solvent.

Next, the present invention is explained referring to the figures.

Figure 1 is an illustrative diagram showing a melt-spinning apparatus of an embodiment of the present invention. (1) is a spinneret, (2) and (3) are cylindrical spray equipments (ring-shaped sprays) that are installed within 10 cm right under the spinneret surface and spray a gas from an outer peripheral direction toward the center direction of the spinneret, and they have a two-stage spray (hereinafter, the upper spray (2) is referred as a first spray, and the lower spray (3) is referred as a second spray). (4) is a yarn spun from the spinneret (1). (5) is a support point guide and is positioned at the upstream from an oil treating apparatus (6) and an initial drawing roller

(hereinafter, referred as a first drawing roller) (7) for prescribing the yarn speed. (8) is a second drawing roller being a pair with the first drawing roller (7). (9) is an interlace nozzle or [illegible] nozzle. (10) is a traverse support point guide, and (11) is a take-up reel. Figure 2 shows the lower surface of the spinneret (1), and spinneret holes (12) are arranged in a ring-shaped hole array zone being prescribed by the minimum hole array diameter (D1) and the maximum hole array diameter (D2).

As long as the conventional ordinary spinning method is adopted, spinning of uniform completely continuous ultra-fine fibers was very difficult due to surface tension, etc., of the polymer, however with the adoption of the present invention, intended completely continuous ultra-fine fibers of a polyester multifilament with a single yarn size of 0.5 denier or less can be simply obtained. Its mechanism has not been clearly elucidated up to now, however it is basically considered that the mechanism is a subtle combination of three factors of swelling, surface tension, and drop speed (drawing speed) of the polyester melt polymer right under the spinneret holes. However, if the number of filament of the spun yarns (4) is increased, problems such as end breakage due to an interaction/3 of an accompanying gas flow being generated in the vicinity of

each filament of the spun yarns, temperature variation of a cooling atmosphere, and difference in cooling, ultra miniaturization, and solidification behaviors being generated by the difference of filament positions such as outer peripheral part and central part are generated in addition to the above-mentioned three basic factors. In other words, removing the tension, cooling, and speed difference between the filaments and ideal cooling, ultra miniaturization, and solidification must be considered as important factors in the industrialization.

Next, the present invention is explained in detail.

The relationship among the spinneret hole diameter $D(\text{mm})$, the amount Q of discharge per each spinneret hole (g/min), and the yarn drawing speed (V) (m/min) and the single yarn size d of the fibers being obtained and the spinning draft V/V_0 are shown by the following equations.

$$d = 9000Q/V \quad (4)$$

$$V/V_0 = \varepsilon \rho D^2 V / 4Q \quad (5)$$

However, V_0 is a discharge rate of a melt polymer being discharged from the spinneret holes (12).

$$V_0 = 4Q/\varepsilon \rho D^2 \quad (\text{m/min})$$

ρ : Density of the melt polymer being discharged (g/cm^3)

As seen from the equation (4), in order to obtain the ultra-fine fibers, it is necessary to largely raise the drawing speed (V)

of the yarns or to decrease the amount (Q) of melt polymer being discharged from each hole of the spinneret holes (12). The manufacture of the ultra-fine fibers by raising only the drawing speed (V) of the yarns (4) is preferable in terms of productivity, however there are various problems such as facility expenses and spinnability. In other words, since the maximum rolling speed of the current take-up reel on the market is 6,000 m/min, a rolling production is impossible at a speed higher than that, and even if a high-speed take-up reel is developed, the facility expenses are enormous.

Furthermore, in case the ultra-fine fibers are obtained by raising only the drawing speed (V) of the yarns while maintaining the amount (Q) of discharge per each hole of the spinneret holes (12) at a conventional level, as seen from the equation (5), it is necessary to raise the spinning draft (V/V_0). As a result, the accompanying gas flow in the vicinity of the spun yarns (4) is extremely increased, and the atmosphere under the spinneret (1) is very disturbed, so that end breakage and cooling irregularity are caused. At the drawing speed (that is, $V = 5,500$ m/min at $Q = 0.45$ g/min) corresponding to the single yarn size of 0.7 denier, a stable spinning is impossible. Next, if the amount (Q) of melt high-molecular polymer being discharged from each hole of the spinneret holes (12) is

decreased, as seen from the equation (4), finer fibers are preferably obtained. However, if the amount (Q) of discharge is slowly lowered by the spinneret (1) with an ordinary spinneret hole diameter (0.25-1.0 mm), the spun yarns (4) are in [illegible] state, so that the yarns are very unstable, thereby being unable to obtain uniform ultra-fine fibers.

Accordingly, these inventors earnestly researched to enable a stable spinning by lowering the amount (Q) of discharge per each hole of the spinneret holes (12) through the elimination of the generation of [illegible] state. As a result, it was discovered that if the spinneret hole diameter (D) was decreased to 0.20 mm or less, the spun yarns (4) were not in [illegible] state and a stable good spinning was possible. In order to obtain ultra-finer fibers, as mentioned above, it is preferable to decrease the amount (Q) of discharge per each hole of the spinneret holes (12) and to raise the drawing speed (V), however as seen from the equation (5), the spinning draft (V/V_0) is extremely increased, so that the draft cut is generated in the spun yarns (4) and a continuous rolling is difficult. However, with the decrease of the spinneret hole diameter (D), since the amount (Q) of melt polymer being discharged from each hole of said spinneret holes (12) can also be decreased, ultra-fine fibers can be obtained by drawing at the drawing speed (V) proportional

to the amount (Q) of discharge, that is, at $18 \times 10^3 \times Q$ (m/min) or more, even without applying a super high-speed drawing. On the contrary, if the spinneret hole diameter (D) and the amount (Q) of discharge per each hole of said spinneret holes (12) are set to the range of the present invention and the drawing speed (V) is lower than $18 \times 10^3 \times Q$ (m/min), since the spinning draft is small and the single yarn size is large at a low drawing tension, cooling is insufficient, so that end breakage and fusion between the yarns are generated. Thereby, yarns being provided for stretching cannot be obtained.

Therefore, spinning at the spinneret hole diameter (D) of 0.20 mm or less and the amount (Q) of high-molecular polymer /4 being discharged per each hole of said spinneret holes (12) of 0.15 g/min or less and drawing at the drawing speed (V) of $18 \times 10^3 \times Q$ (m/min) are essential conditions for manufacturing an intended ultra-fine fibers of the present invention.

On the other hand, in consideration of practical yarn characteristics, workability, and productivity, there is a lower limit in the total denier of the spun yarns, and it is necessary to increase of the number of filaments with the decrease of the single yarn denier. Therefore, for industrialization, it is absolutely necessary to solve the problems being generated in

the multifilament formation such as productivity, end breakage, single yarn irregularity, and workability.

According to the method of the present invention, as mentioned above, since spinning is carried out at a low amount of single hole discharge and a high drawing speed, the miniaturization and solidification of the spun yarns is rapidly advanced and completed at a distance within about 25 cm from the surface of the spinneret (1). Thus, it is most important to strictly control the atmosphere temperature and the gas flow in the vicinity of a number of filaments until the spun yarns (4) are solidified from the surface of the spinneret (1). However, as long as the spinneret in which a number of spinneret holes of 100 holes or more are arranged in a hole array zone in which K of the above-mentioned equation (1) is greater than 0.25, even if the atmosphere temperature and the gas flow in the vicinity of the yarns are adjusted, a difference in the cooling rate is caused between the outer peripheral part and the central part of the spun yarns, so that end breakage and size difference between the filaments are increased. Thereby, high-quality ultra-fine fibers cannot be stably manufactured. In an extreme case, if the atmosphere temperature is applied to the vicinity of the solidification point of the spun yarns, the atmosphere temperature of the central part is 50-100°C higher than the

temperature of the outer peripheral part, and the solidification point of the yarns at the central part is considerably shifted to the downstream from the solidification point of the yarns at the outer peripheral part. Thereby, a difference is caused in the tension and the speed between the yarns until the miniaturization and solidification and yarn shaking, fusion, and end breakage are frequently caused by the interaction of the accompanying gas flow being generated in the vicinity of the yarns.

These inventors also researched these problems in earnest, and as a result, it was discovered that these problems could be solved by improving the method for arranging the spinneret holes and the method for cooling the spun yarns in an area within 10 cm right under the spinneret surface. In other words, in order to make the miniaturization and solidification behaviors of the polyester multifilament spun uniform, the arrangement of the spinneret holes (12) on the surface of the spinneret (1) is set to a ring shape, the value of K being prescribed in the equation (1) is set to 0-0.25, the amount M of flow (Nl/min) of a gas being blown in the central direction of the spinneret from the outer peripheral direction in the area within 10 cm right under the surface of the spinneret (1) is prescribed by the equation (2), and the temperature T ($^{\circ}\text{C}$) of the gas atmosphere in the

vicinity of the spun yarns is adjusted within the range of the equation (3). Thereby, completely continuous ultra-fine yarns of a high-quality polyester multifilament without yarn shaking, fusion, and end breakage can be manufactured. In particular, $K = 0$ in the equation (1) shows that the number of spinneret holes (12) being arranged is one column. If the spinneret is used, the miniaturization and solidification behaviors between each filament are almost uniform, and high-quality continuous ultra-fine yarns with little difference between the single yarns can be stably obtained. In case the value of K is greater than 0.25 for the purpose of a uniform polymer discharge from the entire surface of the spinneret, even if any yarn cooling method is adopted, the difference in the miniaturization and solidification behaviors between the filaments is large, and the arrangement effect of the spinneret holes (12) in a ring shape is lost, so that the above-mentioned problems are caused. Also, even if the spinneret in which the spinneret holes are arranged in a ring shape so that the value of K being prescribed in the equation (1) may be 0-0.25, if the amount M (Nl/min) of gas flow being blown in the central direction from the outer periphery of the spinneret is smaller than the lower limit of the equation (2) in the area within 10 cm right under the spinneret surface, the atmosphere temperature in the central part of the yarns is

higher than that of the outer peripheral part, and the solidification point of the yarns in the central part is considerably deviated to the downstream from the solidification point of the yarns in the outer peripheral part. Thereby, a difference is caused in the tension and speed between the yarns until being miniaturized and solidified, and the accompanying gas flow being generated in the vicinity of the yarns cannot be completely adjusted. As a result, even if the atmosphere temperature is measured at the same position, the temperature variation of about 5-20°C is caused, yarn shaking, fusion, and end breakage are generated, and an aggressive spraying effect can seldom be expected. On the contrary, if the amount M of gas flow being blown is larger than the upper limit of the equation (2), the accompanying gas flow in the vicinity of the yarns and the atmosphere temperature variation being generated right under the spinneret can be suppressed, however since the amount of flow being sprayed is too large, the spun yarns are directly cut by the spraying air, which is not preferable. In other words, basically, it is preferable to set the amount of gas being sprayed on the spun yarns to a value slightly larger than the amount of accompanying gas flow being caused by the spun yarns, and this can be realized by adjusting the amount to the range of the experimental equation (2) being prescribed by a function of

the yarn drawing speed (V) and the total number H of spun filaments.

The spray gas being adopted in the present invention is preferably an air or an inert gas such as nitrogen gas, and $\frac{1}{5}$ the number of spray stage may be one stage, or a multi-stage spraying method such as two stages or more as shown in Figure 1 may also be adopted. In particular, since it is necessary to increase the amount of gas being blown as the total number (H) of spun filaments increases and the drawing speed rises, it is more effective to adopt the spraying method of two stages or more, to set the amount being sprayed from the upper-stage spraying part to an amount smaller than the amount being sprayed from the lower-stage part, to set the temperature of the spraying air at the upper-stage part to a temperature higher than that at the lower-stage part, and to circulate an inert gas such as hot nitrogen gas in the upper-stage part to lengthen the nozzle lifetime. Next, as mentioned above, even if the spinneret that meets the equation (1) and the ring-shaped spraying method that meets the equation (2) are adopted, if the yarn cooling rate (the atmosphere temperature in the vicinity of the yarns) in the area within 10 cm right under the spinneret is inappropriate, that is, if the yarn cooling rate is fast, the draft cut is frequently caused. On the contrary, if the cooling

rate is slow, the tension of the yarns is lowered, and yarn shaking, fusion, and draw resonance phenomenon are generated, so that continuous ultra-fine yarns of a high-quality polyester multifilament cannot be obtained.

Accordingly, these inventors also researched the atmosphere temperature in the vicinity of the yarns right under the spinneret (the temperature obtained by measuring the temperature of the gas separated at 5 mm from the yarns by a CA thermocouple of 0.25 mm \varnothing) in earnest to manufacture the continuous ultra-fine yarns of the high-quality polyester multifilament. As a result, this could be realized by adjusting the temperature to the range of the above-mentioned equation (3). In order to ideally (without causing yarn shaking, fusion, draft cut, [illegible] shape) uniformly cool, miniaturize, and solidify the spun polyester multifilament, it is preferable to set the spinning tension at the solidification point to 0.5-1.0 g/d. For this purpose, it is necessary to adjust the cooling, miniaturization, and solidification rate of the spun yarns by changing the atmosphere temperature right under the spinneret by the degree of polymerization (η) (shown by the relative viscosity in the present invention) of the spun polyester. In other words, if the atmosphere temperature $T(^{\circ}\text{C})$ in the vicinity of the spun yarns right under the spinneret is set to a temperature lower

than the lower limit temperature of the equation (3) for the degree of polymerization (η) of the polyester polymer being used, since rapid yarn miniaturization and solidification are caused, the draft cut is caused, so that intended yarns cannot be obtained. On the contrary, if the temperature is too higher than the upper limit temperature of the equation (3), the cooling, miniaturization, and solidification of the spun yarns are [illegible], the spinning tension is lowered, and yarn shaking, fusion, [illegible], etc., are caused, so that continuous ultra-fine fibers of a high-quality polyester multifilament cannot be stably obtained.

The polyester constituting the polyester yarns in the present invention is a polyester in which at least 70% of the polyester constitution unit is polyethylene terephthalate.

The ultra-fine fibers of the high-quality polyester multifilament obtained by the method of the present invention have very excellent shape cohesion and adhesion. Its properties can be used in the prevention of a hem rise of shirts and an alternative use of a surface fastener, and it can also be used in a fitted overlapping of [illegible], and the fibers are adhered to the human skin, which are unknown special properties.

Furthermore, according to the method of the present invention, since completely continuous ultra-fine fibers can be

obtained, they may also be used as they are, and desired ultra-fine fibers having various fiber performances can also be formed by stretching using a stretcher, like conventional fibers. In particular, its stretch performance is superior to that of the ultra-fine fibers obtained by any manufacturing methods. Also, the present invention is industrially very excellent, and ultra-fine fibers of a completely continuous polyester polymer are formed. However, it is not necessary to remove one component in a solvent, unlike a sea-island fiber dissolution method, and the fibers can be treated similarly to ordinary unstretched yarns or stretched yarns. In other words, these fibers may also be used alone, however they can also be mixed with other thick fibers. Furthermore, as a result, these fibers can give remarkable improvements in terms of corrosion, marked fitness, shape cohesion, lightness, thinness, drape characteristic, and handling.

Next, the present invention is explained in detail by application examples, however the present invention is not limited to these application examples.

Application Example 1

A polyethylene terephthalate with a relative viscosity $\eta = 1.38$ was dissolved by heating it at a spinning temperature (surface temperature of a spinneret) of 285°C using a melt

spinning machine shown in Figure 1 and spun while changing the/6 amount Q (g/min) of discharge per each hole of the spinneret holes (12) and the speed (m/min) of the drawing rollers (7) and (8), so that a package was prepared. The spray used at that time was a cylindrical two-stage type spray. In the first spray, a hot nitrogen gas was sprayed from a spray surface with an inner surface of 110 mm \varnothing and a width of 25 mm, and in the second spray, an air at 50°C was sprayed from a spray surface with an inner diameter of 110 mm \varnothing and a width of 50 mm at the position right under the first spray. Also, the amount M of spray air (Nl/min) and the temperature of the hot nitrogen gas of the first spray were adjusted so that the equations (2) and (3) might be met.

The results are shown in Table II.

Table I

| | |
|---|---|
| Spinneret diameter | 90 mm |
| Innermost peripheral hole diameter (D1) | 69 mm |
| Outermost peripheral hole diameter (D2) | 73 mm |
| Number of hole columns | 2 columns |
| Total number of spinneret holes (H) | 240 |
| Spinneret hole diameter (D) | 0.30 mm, 0.20 mm, 0.10 mm, and 0.05 mm |
| K value | 0.058 |

Table II

| | | 1. 2. 3. 4. | | | | | | | | | |
|-----|------------------|-------------|------|-------|----------------------------|-------------|----------------------------|-------------|----------------------------|-------------|----------------------------|
| | | 表 2 续 | | | | | | | | | |
| | A | D | Q | V | 第 1 次付 | | 第 2 次付 | | 第 3 次付 | | |
| | | | | | 風 量 (N ₂ /分) | 溫 度 (°C) | 風 量 (N ₂ /分) | 溫 度 (°C) | 風 量 (N ₂ /分) | 溫 度 (°C) | 風 量 (N ₂ /分) |
| 10. | 比 單 例 | 1 | 0.30 | 645 | 3200 | 5.40 | 5.160 | 7.210 | 13.8 | 8.9 | 9.45 |
| | | 2 | " | " | 5500 | 50 | 170 | 300 | 0.7 | 5.1 | 4.3 |
| | | 3 | " | 0.25 | 3200 | 40 | " | 210 | 0.7 | 9.2 | 6.0 |
| | | 4 | " | 0.15 | 3250 | 30 | " | 170 | 0.6 | 12.3 | 8.1 |
| | | 5 | " | 0.10 | 1600 | 20 | 175 | 160 | 0.5 | 16.5 | 12.2 |
| 11. | 本 驗 明 法 | 6 | 0.20 | 0.15 | 4500 | 80 | 185 | 240 | 0.3 | 2.9 | 2.0 |
| | | 7 | " | 0.10 | " | " | " | " | 0.2 | 1.5 | 1.2 |
| | | 8 | " | 0.075 | " | " | " | " | 0.125 | 1.7 | 1.3 |
| 12. | 比 較 例 | 9 | 0.10 | " | 1125 | 20 | 170 | 130 | 0.6 | 14.5 | 12.4 |
| | | 10 | " | 0.25 | 5625 | 50 | " | 306 | 0.4 | 6.4 | 1.1 |
| 13. | 本 驗 明 法 | 11 | " | 0.10 | 4500 | 80 | 185 | 240 | 0.2 | 1.2 | 0.9 |
| | | 12 | " | 0.075 | 3375 | 60 | 180 | 220 | 0.2 | 1.0 | 0.8 |
| | | 13 | " | 0.05 | " | " | 185 | " | 0.12 | 0.7 | 0.6 |
| | | 14 | 0.05 | " | 4500 | 70 | 190 | 240 | 0.10 | 0.6 | 0.5 |

(TABLE II, cont.)

| | | 18. | | 19. | |
|-----|-------|-----------------|------------------|-----|--|
| | | 原 糸 綫 子 | 低 伸 縮 子 | | |
| 14. | 比 較 例 | 1 糸徑九中中大 | 20. 不良 DR = 4.32 | 24. | |
| | | 2 略 + 切断発生 | | 25. | |
| | | 3 糸徑九中中大 | 不良 DR = 2.32 | | |
| | | 4 略 + 切断発生 | | | |
| | | 5 略 + 切断発生 | | | |
| 15. | 不良例 | 6 良 好 | 良好 DR = 1.20 | | |
| | | 7 21. | | 26. | |
| 16. | 比 較 例 | 8 | | | |
| | | 9 略 + 切断発生 | 不良 DR = 2.0 | | |
| | | 10 糸徑九大, 略 + 22 | 不良 DR = 1.22 | 27. | |
| | | 11 良 好 | | | |
| 17. | 不良例 | 12 | | | |
| | | 13 23. | | | |
| | | 14 | | | |

28. (D 値: 低伸縮率)

TABLE II KEY:

1. First spray
2. Second spray
3. Constitutional single yarn size (denier)
4. Single yarn irregularity (%)
5. Amount of air (Nl/min)
6. Temperature (°C)
7. Amount of air (Nl/min)

8. Between the single yarns
9. In the single yarn
10. Comparative Example
11. Method of the present invention
12. Comparative Example
13. Method of the present invention
14. Comparative Example
15. Method of the present invention
16. Comparative Example
17. Method of the present invention
18. Spinning state
19. Stretching state
20. Slightly large yarn shaking
Often cut generation
Slightly large yarn shaking
[illegible] state, cut generation
[illegible] state, cut generation
21. Good
22. [illegible] state, fusion
Large yarn shaking, fusion
23. Good
24. Inferior DR = 4.32
25. Inferior DR = 2.33

- 26. Good DR = 1.20
- 27. Inferior DR = 2.0
Inferior DR = 1.33
- 28. (DR: Stretch magnitude)

Sample Nos. 6-8 and 11-14 spun and drawn by the method of/7 the present invention were very stable without spinning [illegible], fusion, and yarn shaking. In particular, in the sample Nos. 11-14 in which the spinneret hole diameter (D) and the amount (Q) of discharge per one spinneret hole were small and drawing was carried out at a high spinning speed, the single yarn size was 0.20 denier or less, and high-quality completely continuous ultra-fine fibers with very little single yarn irregularity could be stably obtained. Also, in sample Nos. 1-3 out of the range of the present invention, since the amount (Q) of discharge per one spinneret hole was large, the cooling and solidification rate was slow, and the yarn shaking was large, the single yarn irregularity was also large, and the stretch performance was inferior. In sample Nos. 4 and 5, since the spinneret hole diameter (D) was large, if the amount (Q) of discharge per one spinneret hole was small, the spun yarns (4) was [illegible] shape right under the spinneret (1), and as the single yarn irregularity was extremely increased and became

severe, cutting was caused, and a continuous drawing was impossible. In sample No. 9, the spinneret hole diameter (D) and the amount (Q) of discharge were in the range of the present invention, however since the drawing rate was low, the tension being exerted on the yarns (4) was low. The spun yarns were unstable and [illegible] shape. Also, due to their low tension, the yarns were also easily shaken, and fusion was generated between the yarns. In sample No. 10, since the amount (Q) of discharge per one spinneret hole at a spinneret hole diameter (D) of 0.10 mm was as high as 0.25 g/min, the cooling and solidification of the spun yarns (4) was slow, and since the spinning draft (V/V_0) was as small as about 210, so that the yarn shaking was large and fusion was caused. The yarns of the sample Nos. 1, 3, 9, and 10 were subjected to a two-yarn doubling stretch by a one-stage stretch using an ordinary stretcher so that the final single yarn size might be 0.30 denier, however fuzzes and cutting were frequently generated during the stretch. In the sample No. 6 to which the method of the present invention was applied, a two-yarn doubling stretch was also carried out by a simple method, and high-quality completely continuous ultra-fine yarns with [illegible] of 120 d/480f, a strength of 5.1 g/d, and a cut stretch of 23% could be obtained without any troubles such as fuzzes and cuts. In the

sample Nos. 7, 8, and 11-14, uniform continuous ultra-fine fibers of 0.20 denier or less were obtained without stretching. Also, as for the irregularity between the single yarns, the diameter ($\bar{2r}$) of 30 pieces of single yarns was measured at random, and the average $\bar{2r}_{\max}$ of 5 pieces with a thick single yarn diameter and the average $\bar{2r}_{\min}$ of 5 pieces with a fine single yarn diameter were calculated. The irregularity was attained from $\bar{2r}_{\max} - \bar{2r}_{\min} / \bar{2r} \times 100$ (however, $\bar{2r}$ is an average single yarn diameter of 30 pieces). As for the irregularity in the single yarn, the single yarn diameter of one single yarn with a length of 50 mm was measured at random at 30 points in the longitudinal direction. The average $\bar{2r}'_{\max}$ of five positions of the thick single yarn diameter and the average $\bar{2r}'_{\min}$ of five positions of the fine single yarn diameter were calculated, and the irregularity was attained from $\bar{2r}'_{\max} - \bar{2r}'_{\min} / \bar{2r}' \times 100$ (however, $\bar{2r}'$ is an average single yarn diameter of 30 points).

Application Example 2

Using the same melt-spinning machine as that of Application Example 1, a polyethylene terephthalate with a relative viscosity $\eta = 1.45$ was dissolved by heating at a spinning temperature of 300°C, and using a spinneret shown in Table III,

drawing is carried out at a drawing rate of 3,500 m/min while changing the amount (Q) of discharge per one spinneret hole. At that time, a hot nitrogen gas at 205°C was sprayed at 70 (Nl/min) on the spun yarns (4) from the first spray, and an air at 75°C was sprayed at a ratio of 280 (Nl/min) from the second spray. Then, the yarns were cooled and solidified. The spinning conditions and the results are shown in Table IV.

Table III

| | |
|---|-----------|
| Spinneret diameter | 90 mm |
| Innermost peripheral hole diameter (D1) | 65 mm |
| Outermost peripheral hole diameter (D2) | 73 mm |
| Number of hole columns | 3 columns |
| Total number of spinneret holes (H) | 350 |
| Spinneret hole diameter (D) | 0.10 mm |
| K value | 0.123 |

Table IV

| | | | | | | | | |
|----|------|---|-------|------------------------|-----------------|-----|-------------|----|
| | | | | 1. | 2. | | 3. | |
| | | | | 第 4 款 | | | | |
| | | | | 繰取率 糸繰量 (g/100m) | 単 糸 距 (%) 4. | 5. | 繰 糸 回 子 | |
| 6. | 比較例 | 1 | 0.25 | 0.64 | 9.8 | 7.7 | 糸繰れ大、細糸発生 | 8. |
| | | 2 | 0.20 | 0.51 | 8.6 | 5.3 | 糸繰れ大、時々細糸発生 | |
| | | 3 | 0.15 | 0.39 | 8.8 | 3.3 | 糸繰れやや大、細糸あり | |
| 7. | 本発明法 | 4 | 0.10 | 0.26 | 8.4 | 2.5 | 糸繰れ小、 | 9. |
| | | 5 | 0.075 | 0.19 | 2.9 | 2.2 | ' | |
| | | 6 | 0.05 | 0.13 | 2.0 | 1.8 | ' | |

1. Single yarn size (denier)
2. Single yarn irregularity (%)
3. Spinning state
4. Between single yarns
5. In single yarn
6. Comparative Example
7. Method of the present invention
8. Large yarn shaking, [illegible] yarn generation
Large yarn shaking, often [illegible] generation
9. Slightly large yarn shaking, [illegible]
Small yarn shaking

In sample Nos. 1 and 2, since the amount (Q) of discharge per one spinneret hole is high, even if it was drawn out at a

draw rate of 3,500 min/min, the single yarn size was not considerably small. Furthermore, since the single yarn size was large, the cooling and solidification of the spun yarns (4) was slow, and since the spinning draft was small (the spinning draft of Nos. 1 and 2 was 106 and 133, respectively), the tension being exerted on the spun yarns (4) was low. The spinning yarns were unstable, yarn shaking and fusion were easily caused, and only yarns with a large single yarn irregularity were obtained. In sample Nos. 3-6, the method of the present invention was adopted, and the spinning state was good. In particular, in the yarns of the sample Nos. 3 and 4 were stretched 1.95 and 1.3 /8 times, respectively by an ordinary one-stage stretch, and high-quality completely continuous polyester ultra-fine yarns with a final single yarn size of 0.20 denier without fuzzes and end breakage could be obtained. Sample Nos. 1 and 2 were also stretched 3.2 and 2.6 times (a final single yarn size of 0.20 denier), respectively by a similar method, however fuzzes and end breakage were frequently generated, so that a continuous stretch was impossible. Also, at that time, the atmosphere temperature $T(^{\circ}\text{C})$ in the vicinity of the yarns (the position of 5 mm from the yarns) right under the spinneret surface was measured using a CA thermocouple of 0.25 mm \varnothing . The results are shown in Table V.

Table V: Atmosphere temperature $T(^{\circ}\text{C})$ in the vicinity of yarns

2.

| 1. | 口金面下 距離 L mm | 0 | 1 | 2 | 3 | 4 | 5 | 7 | 10 |
|----|--------------------|-----|-----|-----|-----|-----|-----|-----|----|
| | 試料 No. | | | | | | | | |
| | 1 | 240 | 271 | 228 | 180 | 134 | 111 | 104 | 93 |
| | 2 | " | 267 | 222 | 172 | 126 | 107 | 100 | 90 |
| | 3 | " | 263 | 217 | 165 | 122 | 103 | 97 | 88 |
| | 4 | " | 259 | 213 | 159 | 117 | 98 | 93 | 85 |
| | 5 | " | 256 | 208 | 155 | 113 | 86 | 81 | 83 |
| | 6 | " | 252 | 205 | 151 | 110 | 93 | 89 | 82 |

1. Distance L under the spinneret surface (cm)

2. Sample No.

The sample Nos. 1-6 are within the temperature range that meets the equation (3). Furthermore, the temperature variation at each measured point was within $\pm 1^{\circ}\text{C}$ and was stable.

Application Example 3

Using a melt-spinning machine in which a cylindrical spray right under a spinneret was a one-stage spray (at the position of 30 mm right under the spinneret, an inner diameter of 110 mm \varnothing and a spray surface width of 50 mm), a polyethylene terephthalate with a relative viscosity $\eta = 1.30$ was dissolved by heating at a spinning temperature of 270°C . Using a spinneret

shown in Table VI, the amount (Q) of discharge per one spinneret hole was set to 0.075 g/min so that the average size of the single yarns might be 0.15 denier, and the speed was set to 4,500 m/min. In this state, a drawn package was obtained. Also, an air heated at 115°C was sprayed at 200 (Nl/min) and 300 (Nl/min) at the total number of spinneret holes of H = 120 and 240 so that the atmosphere temperature in the vicinity of the yarns right under the spinneret. At that time, the spinning state and the single yarn irregularity are shown in Table VII.

Table VI

1.

第 6 表

| | 射出口金 | | | | | |
|------------|------|------------------|-------|-------|-------|-------|
| | | A | B | C | D | E |
| 射出口金面数 | | 90mm | 90 | 90 | 90 | 90 |
| 最内周孔徑 (D1) | | 73mm | 49 | 60 | 55 | 37 |
| 最外周孔徑 (Dt) | | 73 | 73 | 73 | 73 | 73 |
| 2. 孔列数 | | 1 列 ³ | 1 | 3 | 3 | 5 |
| 口金孔数 (H) | | 120 | 240 | 240 | 240 | 240 |
| 口金孔徑 (D) | | 0.10mm | 0.10 | 0.10 | 0.10 | 0.10 |
| K 值 | | 0 | 0.05% | 0.216 | 0.327 | 0.973 |

1. Spinneret

2. Spinneret diameter

Innermost peripheral hole diameter (D1)

Outermost peripheral hole diameter (D2)

Number of hole columns

Total number of spinneret holes (H)

Spinneret hole diameter (D)

K value

3. One column

Table VII

| 第 7 例 | | 1. | 2. | 3. | |
|-------|---|----------|-------------|-------|-----------------------------|
| | A | 細糸 口金 | 單 糸 變 異 (%) | | 新 糸 變 異 子 |
| | | | 單 糸 間 | 單 糸 內 | |
| 6. | 1 | A | 4.45 | 5.04 | 非常良好 8. |
| | 2 | B | 11 | 8.7 | 良 好 |
| | 3 | C | 22 | 23 | 糸變異中々大 |
| 7. | 4 | D | 68 | 61 | 內周部糸變異大, 時々斷糸多し 9. |
| | 5 | E | 157 | 146 | 內周部糸變異大且く, 斷糸, 切斷多量, 流注結束不可 |

1. Spinneret

2. Single yarn irregularity (%)

3. Spinning state

4. Between single yarns

5. In single yarn

6. Method of the present invention

7. Comparative Example

8. Very good

Good

Slightly large yarn shaking

9. Large yarn shaking at the inner peripheral part

Often

Large yarn shaking at the inner peripheral part,

[illegible], frequency end breakage, and impossible

continuous spinning

In sample Nos. 1-3, since the spinneret of the method of the present invention was used, the spinning state was good, the single yarn irregularity was also small, and high-quality continuous ultra-fine fibers were obtained. In particular, in the sample Nos. 1 and 2, since the number of hole columns was one column or two column and the value of K was very small, a uniform cooling was possible, the miniaturization and solidification behaviors between the yarns was uniform, the yarns were seldom shaken, and high-quality continuous ultra-fine yarns with very small single yarn irregularity could be stably obtained. In sample Nos. 4 and 5, a cooling difference was caused between the yarns, a uniform miniaturization and solidification was difficult, the spinning state was also unstable, and fusion and end breakages were often caused.

In particular, in the sample No. 5 using an ordinary spinneret, the end breakage was frequently caused (the yarn shaking at the central part was large, and the fusion was frequently caused), and continuous yarns could not be sampled.

Application Example 4

Using the same melt-spinning machine as that of Application Example 1, a polyethylene terephthalate with a relative viscosity $\eta = 1.36$ was dissolved by heating at a spinning temperature of 280°C . Using a spinneret shown in B of Table VI, while variously changing the amount of flow, the amount (Q) of discharge per one spinneret hole was set to 0.075 g/min , a hot/9 nitrogen gas was sprayed from the first spray, and an air at 50°C was sprayed from the second spray so that the atmosphere temperature $T(^{\circ}\text{C})$ in the vicinity of the spun yarns might meet the equation (1). Thus, a package was prepared at a constant drawing rate of $4,500 \text{ m/min}$. At that time, the spinning state and the single yarn irregularity are shown in Table VIII.

Table VIII

| | | 1. | | 2. | | 3. | | 4. | |
|-----|------|----------------|------------|----------------|------------|---------|-----------------|------|-----|
| | | 第1吹付 | | 第2吹付 | | 单糸疵 (%) | | 紡糸様子 | |
| | | 風量 (Nl/min) | 温度 (°C) | 風量 (Nl/min) | 温度 (°C) | 单糸間 | 单糸内 | | |
| 10. | 比較例 | 1 | 50 175 | 700 | 8.3 | 9.7 | 疵多し発生、糸切れ大、時々切断 | | 13. |
| | 本発明法 | 2 | 50 150 | 150 | 2.4 | 1.8 | 良 好 | | 14. |
| 11. | 比較例 | 3 | 80 185 | 240 | 0.8 | 0.6 | 非常に安定し良好 | | |
| | 本発明法 | 4 | 100 185 | 100 | 1.5 | 1.1 | 良 好 | | |
| 12. | 比較例 | 5 | 125 185 | 175 | 2.5 | 2.0 | 糸切れ大、時々切断 | | 15. |

1. First spray
2. Second spray
3. Single yarn irregularity (%)
4. Spinning state
5. Amount of air (Nl/min)
6. Temperature (°C)
7. Amount of air (Nl/min)
8. Between single yarns
9. In single yarn
10. Comparative Example
11. Method of the present invention
12. Comparative Example

13. [illegible] generation, large yarn shaking, and often end breakage
14. Good
[illegible] stable and good
Good
15. Large yarn shaking, often end breakage

Sample Nos. 2-4 were adjusted between 5-80 mm (spraying surface width = $25 + 50 = 75$ mm) right under the spinneret surface so that the amount M of air being sprayed (the amount of nitrogen gas + the amount of air) might meet the equation (3), and the single yarn irregularity was small, and very stable, continuous spinning and sampling were possible. In sample No. 1, since the amount M of air being sprayed was as small as 120 (Nl/min), the accompanying air flow being generated by the spurn yarns cannot be rectified, and the atmosphere temperature right under the spinneret surface was varied by $\pm 5^{\circ}\text{C}$ or higher and became [illegible] shape. At the same time, the yarns were largely shaken, and their continuous spinning was impossible. Also, in sample No. 5, since the amount M of air being sprayed was larger than the amount of air being generated right under the spinneret surface, the air flow right under the spinneret was disturbed by the spraying [illegible], and the yarns were

largely shaken. Sometimes, end breakage was caused, and the yarns could not be continuous spun. Also, it is not preferable to spray the hot air on the spun yarns more than is necessary in terms of [illegible] of the spinning [illegible] and economical efficiency.

Application Example 5

Using the same melt-spinning machine and spinneret as those of Application Example 4, a polyethylene terephthalate/isophthalate copolymer polyester (10 mol% isophthalate component) with a relative viscosity $\eta = 1.38$ was dissolved by heating at a spinning temperature of 283°C, and the amount (Q) of discharge per one spinneret hole was set to 0.075 g/min. In order to change the yarn cooling conditions right under the spinneret, the amount of gas being sprayed from the first spray and the second spray was fixed to 320 (Nl/min), and the temperature and the amount of nitrogen as being sprayed from the first spray and air being sprayed from the second spray were variously changed. A package was prepared at a constant drawing rate of 4,500 m/min. At that time, the atmosphere temperature T(°C) in the vicinity of the spun yarns right under the spinneret surface, the spinning state, and the single yarn irregularity are shown in Tables IX and X.

Table IX

1. 2.

第 9 表、

| | K | 第 1 吹付 | | 第 2 吹付 | | T (°C) | | | | | | | |
|----|-----|--------------|----------|--------------|-----------|----------------------|-------|-----|-----|-----|-----|-----|------|
| | | 風量 Nl/min | 温度 °C | 風量 Nl/min | 温度 °C | 5. 紡糸口金面からの距離 L (mm) | | | | | | | |
| | | | | | | | | | | | | | |
| | | | | | | 0mm | 1mm | 2mm | 3mm | 4mm | 5mm | 7mm | 10mm |
| 6. | 比較例 | 1 | 3. 50 | 4. 250 | 3. 270 | 4. 130 | 208 | 262 | 250 | 222 | 198 | 176 | 151 |
| | 本発明 | 2 | 50 | 210 | 270 | 75 | 208 | 242 | 215 | 162 | 120 | 114 | 102 |
| 7. | 例 | 3 | 50 | 240 | 270 | 20 | 283 | 255 | 220 | 160 | 92 | 76 | 54 |
| 8. | 比較例 | 4 | 80 | 140 | 240 | 20 | (275) | 171 | 114 | 66 | 52 | 50 | 47 |

1. First spray
2. Second spray
3. Amount of air, Nl/min
4. Temperature, °C
5. Distance L (mm) from the spinneret surface
6. Comparative Example
7. Method of the present invention
8. Comparative Example

Table X

| | | | | | |
|--------|---------|---|-----------|-----------|---|
| | | | 1. | 2. | |
| 図 10 表 | | | 單 糸 疵 (%) | | |
| | | | 單 糸 間 | 單 糸 内 | 紡 糸 状 子 |
| 5. | 比 較 例 | 1 | 5.8 3. | 4.1 4. | 糸 揺 れ 大, 密 着 糸 融 生 切 断 多 発 し 通 続 紡 糸 不 可 |
| 6. | 本 発 明 法 | 2 | 1.0 | 0.9 | 良 好 |
| | | 3 | 1.4 | 1.1 | 良 好 |
| 7. | 比 較 例 | 4 | 1.7 | 1.5 | ド ラ フ ト 切 断 し, 通 続 発 取 不 可 |
| | | | | | 10. |

1. Single yarn irregularity (%)
2. Spinning state
3. Between single yarns
4. In single yarn
5. Comparative Example
6. Method of the present invention
7. Comparative Example
8. Large yarn shaking, yarn fusion, frequent end breakage,
and impossible continuous spinning
9. Good
Good
10. Draft cut, impossible continuous spinning

In sample No. 1, since the temperature of nitrogen and air of the first spray and the second spray was too high, the atmosphere temperature in the vicinity of the yarns after 2 cm right under the spinneret surface was higher than the upper limit temperature of the equation (1), and the tension of the spun yarns was extremely lowered (0.3 g/d or less). End shaking was severe, and fusion and cut were frequently generated. In sample No. 4, on the contrary, since the temperature of the nitrogen gas of the first gas was low, the spinneret surface was lowered down to 275°C, and the atmosphere temperature in the vicinity of the yarns at about 1-3 cm right under the spinneret was the lower limit temperature or lower of the equation (2)./10 A complete draft cut was caused, and a continuous sampling was impossible. Also, sample Nos. 2 and 3 were based on the method of the present invention. The single yarn irregularity was small, and the spinning state was very good.

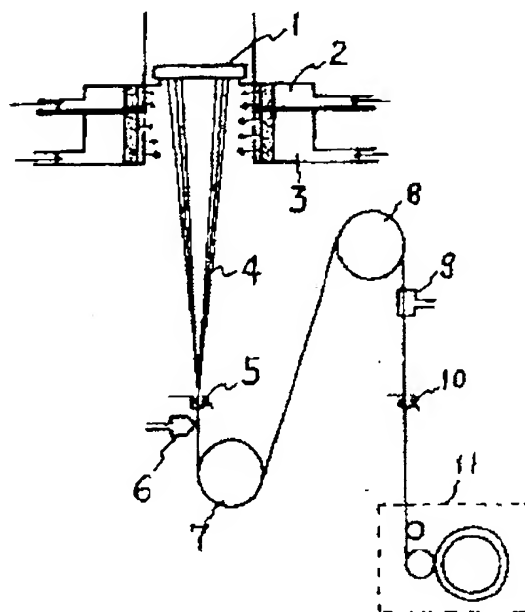
4. Brief description of the figures

Figure 1 is an illustrative diagram showing a melt-spinning apparatus in an application example of the present invention. Figure 2 is a bottom view showing a spinneret.

1 Spinneret

- 2 First spray
- 3 Second spray
- 4 Spinneret

第 1 圖



第 2 圖

